

(12) INTERNATIONAL APPLICATION PUBLISHED UNDER THE PATENT COOPERATION TREATY (PCT)

(19) World Intellectual Property Organization
International Bureau



(43) International Publication Date
27 December 2002 (27.12.2002)

PCT

(10) International Publication Number
WO 02/102329 A1

(51) International Patent Classification⁷: A61K 7/00, 7/04

(74) Agents: STEFFEY, Charles, E. et al.; Schwegman, Lundberg, Woessner & Kluth, P.O. Box 2938, Minneapolis, MN 55402 (US).

(21) International Application Number: PCT/US02/16177

(22) International Filing Date: 21 May 2002 (21.05.2002)

(81) Designated States (national): AE, AG, AL, AM, AT, AU, AZ, BA, BB, BG, BR, BY, BZ, CA, CH, CN, CO, CR, CU, CZ, DE, DK, DM, DZ, EC, EE, ES, FI, GB, GD, GE, GH, GM, HR, HU, ID, IL, IN, IS, JP, KE, KG, KP, KR, KZ, LC, LK, LR, LS, LT, LU, LV, MA, MD, MG, MK, MN, MW, MX, MZ, NO, NZ, OM, PH, PL, PT, RO, RU, SD, SE, SG, SI, SK, SL, TJ, TM, TN, TR, TT, TZ, UA, UG, US, UZ, VN, YU, ZA, ZM, ZW.

(25) Filing Language: English

(26) Publication Language: English

(30) Priority Data:
60/292,424 21 May 2001 (21.05.2001) US

(84) Designated States (regional): ARIPO patent (GH, GM, KE, LS, MW, MZ, SD, SL, SZ, TZ, UG, ZM, ZW), Eurasian patent (AM, AZ, BY, KG, KZ, MD, RU, TJ, TM), European patent (AT, BE, CH, CY, DE, DK, ES, FI, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE, TR), OAPI patent (BF, BJ, CF, CG, CI, CM, GA, GN, GQ, GW, ML, MR, NE, SN, TD, TG).

(71) Applicants and

(72) Inventors: AMATO, Steven, W. [US/US]; 330 Brook Avenue, North Plainfield, NJ 07060 (US). BARONE, Salvatore [US/US]; Apartment 11, 160 Devon Loop, Staten Island, NY 10314 (US). HOYTE, Wayne, M. [US/US]; 17 Buttonwood Drive, Parlin, NJ 08859 (US). NARANJO, Alexander [US/US]; 19 Central Avenue, Mine Hill, NJ 07803 (US). PATEL, Shirish [US/US]; 429 68th Street, Guttenberg, NJ 07093 (US).

Published:

— with international search report

For two-letter codes and other abbreviations, refer to the "Guidance Notes on Codes and Abbreviations" appearing at the beginning of each regular issue of the PCT Gazette.

WO 02/102329 A1

(54) Title: COSMETIC FILMS

(57) Abstract: The present invention includes a cosmetic film. The film includes butylphthalimide isopropylphthalimide, one or more primary film forming polymers and one or more secondary film forming resins.

COSMETIC FILMS

5

Technical Field

The present invention relates to compositions useful as cosmetic films formed from plasticizers alternative to replace phthalates. The compositions herein are particularly useful as coatings for mammalian nails, and are useful as basecoat, topcoat and colored nail polish compositions.

10

Background of the Invention

Nail polish compositions have long been promoted as having long wear, good adhesion, and/or resistance to chipping. While some nail polish compositions have provided better wear than others, a need remains for nail 15 polish compositions providing long wear and employing plasticizers other than phthalate compounds.

Nail polish has, over the decades, been developed to have multi-faceted functionality. In addition to having functionality, polish must be aesthetically pleasing when applied to fingernails and toenails in order to provide a desired 20 color to the nails. The polish must also be resistant to chipping, cracking, splintering and peeling when subjected to a wide range of environments. Over the years, nail polishes have been developed which have these properties to varying degrees.

Nail polishes have typically included pigments and dyes that are 25 suspended in a viscous matrix. The viscous matrix typically includes a film forming component and a plasticizer, namely dibutyl phthalate. The nail polish has also included an adhesion promoter, a polymeric component formed by the condensation polymerization of formaldehyde or other sulfonamide formaldehyde resin.

30 It has been observed over the years that phthalates and aldehyde condensation product exposure has been undesirable for some users because the users are sensitized to these materials. There has been an effort to replace the polymeric component with other materials that do not cause a sensitization.

However, the replacement materials have produced a nail polish that is deficient in features such as high gloss, defined color, long wear, chip resistance, nail flexibility and adherence.

Summary of the Invention

5 One embodiment of the present invention includes a cosmetic film. The film includes butylphthalimide isopropylphthalimide, one or more primary film forming polymers and one or more secondary film forming resins.

Another embodiment of the present invention includes a nail enamel. The clear nail enamel includes butylphthalimide isopropylphthalimide. The nail 10 enamel also includes a secondary film-forming polymer, nitrocellulose and a polyester resin.

One other nail enamel embodiment of the present invention is a colored nail enamel, that includes butylphthalimide isopropylphthalimide in a concentration of about 1 to 10 percent by weight; ethyl acetate in a concentration of about 15 19 of about 37 to 39 percent by weight; butyl acetate in a concentration of about 26 percent by weight; nitrocellulose in a concentration of about 11 to 14 percent by weight; isopropyl alcohol in a concentration of about 5 to 6 percent by weight; optionally, polyester resin in a concentration of about 10 to 12 percent by weight; optionally, tosylamide/epoxy resin in a concentration of 20 about 8 to 10 percent by weight; stearalkonium hectorite in a concentration of about 0.5 to 2 percent by weight; acrylic copolymer in a concentration of about 1.5 percent by weight; and coloring agents in a concentration effective to produce color.

Another embodiment of the present invention includes a nail enamel that 25 includes a plasticizer and butylphthalimide isopropylphthalimide (BIP) in a concentration effective for imparting to the nail enamel, adhesion, hardness and flexibility that is retained for at least about seven days, when the nail enamel is applied to a nail.

The present invention also includes a colored nail enamel, comprising: 30 ethyl acetate; butyl acetate; nitrocellulose; isopropyl alcohol; tosylamide/epoxy resin; butylphthalimide isopropylphthalimide in a concentration of about 1 to about 10 percent by weight; and coloring agents.

The present invention also includes a method for making a cosmetic film that has a hardness, adhesion, and flexibility, gloss, and long shelf life, comprising:

- 5 providing one or more primary film forming polymers;
- providing one or more secondary film forming resins;
- providing butylphthalimide isopropylphthalimide;
- and blending the primary film forming polymer, secondary film forming resin and butylphthalimide isopropylphthalimide (BIP), wherein the BIP has a weight percent concentration of about 1 to 10 weight percent to make a cosmetic
- 10 film having a hardness, adhesion, flexibility, gloss and long shelf life.

Another embodiment of the present invention is a method for coating nails. The method includes providing a cosmetic film that includes butylphthalimide isopropylphthalimide and applying the cosmetic film to one or more mammalian nail.

- 15 One other embodiment includes a colored nail enamel. The colored nail enamel includes an acetate, nitrocellulose, butylphthalimide isopropylphthalimide, tosylamide/epoxy resin and coloring agents.

- 20 Other embodiments include cosmetic films that comprise butylphthalimide isopropylphthalimide; films that comprise butylphthalimide; and films that comprise isopropylphthalimide.

Detailed Description

- One embodiment of the present invention includes a nail enamel composition that comprises butylphthalimide isopropylphthalimide, BIP. The nail enamel of the present invention is, for some embodiments, free of phthalates and, for some embodiments, free of aldehyde condensation products. The nail enamel composition also includes one or more primary film forming polymers and one or more secondary film forming resins. The nail enamel compositions of the present invention include primary film forming polymers and combinations thereof in an amount ranging from about 5 to 20% by weight, and, for some embodiments, in the range of about 9 to 14% by weight.
- 25
- 30

In addition to the primary film forming polymer and secondary film forming resin, the nail enamel compositions according to the present invention

also include at least one plasticizer to soften and to plasticize the primary film forming polymer. The plasticizer as well as combinations thereof, is in either liquid or solid form.

In its product aspect, embodiments of the present invention provide nail 5 enamel compositions which are suitable for use as base coats, color coats, clear coats and protective top coats while maintaining the desirable characteristics of the nail enamel compositions. The nail enamel compositions display good lastingness and chip resistance, and are safe to use with frequent applications.

The present invention includes a use of butylphthalimide 10 isopropylphthalimide, Pelemol BIP, as a plasticizer in nail enamel compositions. Hereinafter, Pelemol BIP is referred to as BIP. While Pelemol BIP is described herein, it is believed that butylphthalimide and isopropylphthalimide, when obtained separately, can be blended to obtain a plasticizer suitable for use in the nail enamel of the present invention. It is also believed that butylphthalimide 15 and isopropylphthalimide are individually usable as plasticizers in nail enamel compositions.

As used herein, the term, "cosmetic film" refers to a nail enamel usable as a base coat, color coat, clear coat and protective top coat. As used herein, "lacquer," "polish" and "enamel" are used interchangeably.

20 Referred to herein are trade names for materials including, but not limited to, polymers and optional components. The inventors herein do not intend to be limited by materials described and referenced by a certain trade name. Equivalent materials (e.g., those obtained from a different source under a different name or catalog (reference) number) to those referenced by trade name 25 may be substituted and utilized in the methods described and claimed herein. All percentages and ratios are calculated by weight unless otherwise indicated. All percentages are calculated based on the total composition unless otherwise indicated. All component or composition levels are in reference to the active level of that component or composition, and are exclusive of impurities, for 30 example, residual solvents or by-products, which may be present in commercially available sources.

The cosmetic properties of the nail varnish of the present invention include good adhesion to the surface of the nail. In addition, the varnish film is

homogeneous and glossy, and exhibits a degree of flexibility that prevents premature cracking and flaking off the nail. In order to obtain such properties, the varnish film is sufficiently soft. This softness allows the film to withstand impact and deformations of the matrix of the nail. However, the film retains an acceptable degree of gloss and is sufficiently hard to prevent scratches and cracks. Nail enamel embodiments are believed to be resistant to cracking for at least about ten days after application to a nail.

The film-forming polymers used in the lacquer of the present invention are selected from polyurethanes, polyacryls, polymethacryls, cellulosic polymers, styrene-acryl copolymers, polystyrene-polyacryl mixtures, polysiloxanes, polyethers, polyesters, urethane-acryl copolymers, cellulose acetate propionate, siloxane-urethane copolymers, polyurethane-polymethacryl mixtures, silicone-acryl copolymers, vinyl acetate polymers, and mixtures thereof. Exemplary secondary film forming resins usable in the formulations of the present invention include, for example, toluene sulfonamide/epoxy resins, e.g. tosylamide and non-drying alkyd resins, acrylic polymers and copolymers, polyurethane, polyacryls, polymethacryls, cellulosic polymers, styrene-acryl copolymers, polystyrene-polyacryl mixtures, polysiloxanes, polyethers, polyesters, cellulose acetate propionate, urethane-acryl copolymers, siloxane-urethane copolymers, polyurethane-polymethacryl mixtures, silicone-acryl copolymers, vinyl acetate polymers, and mixtures thereof. It is also within the scope of the nail enamel compositions of the present invention to include aldehyde condensation products such as arylsulfonamide formaldehyde resins, specifically toluene sulfonamide formaldehyde resin which is a condensation product of formaldehyde and toluene sulfonamide. These secondary film forming resins are added to the nail enamel compositions of the present invention to strengthen and add acceptable wear characteristics to the primary film forming polymer. In general, the amount of secondary film forming resin ranges from about 2 to 20 percent by weight of the composition, and preferably, about 5 to 12 percent of the composition.

Nitrocellulose is a primary film-forming component in some formulation embodiments of the present invention. A use of low levels of nitrocellulose tends to result in the coated films being easily damaged. On the other hand, a

use of high levels of nitrocellulose results in the coated film being too hard and inflexible, resulting in undesirable peeling and hence poor wear resistance.

Other satisfactory film-forming components include cellulose acetate, cellulose acetate butyrate, cellulose acetate propionate, vinyl polymers, 5 polyurethanes, and mixtures of polyurethanes with cellulose acetate butyrate or with nitrocellulose as well as methacrylate and acrylate type polymers. The film-forming component is present in an amount sufficient to provide a stable film upon the nail following the application of the nail enamel to the nail. In general, amounts of the film-forming component of about 8 to about 40 weight 10 percent for some embodiments, and about 10 to about 15 weight percent for other embodiments, are satisfactory. Examples of nitrocellulose are the so called nitrocellulose RS 1/8 sec., RS 1/4 sec., and nitrocellulose 1/2 sec, and nitrocellulose RS 5-6 sec. and 60-80 sec., which have higher viscosities than the earlier grades. The term "RS" refers to the brand of nitrocellulose with a 15 nitrogen content of about 11.2 to 12.8 percent with solubility in esters, ketones, and glycol ethers manufactured by GreenTree Technologies, previously known as Hercules, Inc. The terms 1/8 sec., 1/2 sec, 5-6 sec, and so forth represent viscosity and refer to the time it takes for a ball to fall a given depth in the material. Nitrocellulose is typically supplied in 70% concentrations, wet with 20 30% ethyl or isopropyl alcohol. As used in the present invention, the percentage of nitrocellulose in a given composition is on a dry basis.

In one embodiment, the resins include Shin-Etsu KP-543 (acrylic-silicone resin), Uniplex 670P (polyester resin), PCI Group Inc. Lacq-Hard LH-75 (polyamide resin).

25 Suitable plasticizers are incorporated in the formulation to provide flexibility while exhibiting the wear and chip resistance and shelf stability needed for the composition. Without intending to be limited by theory, it is believed that plasticizers cause a composition to become more easily deformed. Typically, plasticizers are needed in the compositions of the present invention. 30 The compositions of the present invention may comprise from 0% to about 15%, for some embodiments, from 0% to about 10%, and in some embodiments, from about 0% to about 7%, by weight of the composition, of a plasticizer. One or more plasticizers are optionally added to the present compositions.

In one embodiment, the plasticizers include camphor, castor oil, Unitex Chemical Corporation Uniplex 108 (N-ethyl-o/p-Toluenesulfonamide), Uniplex 125A (di-(2-ethylhexyl) adipate), Uniplex 165 (diisobutyl Adipate), Uniplex 260 (Glyceryl Tribenzoate), Uniplex 552 (Pentaerytritol tetrabenzoate), Phoenix 5 Chemical Inc. Pelemol DIA (diisopropyl adipate), Pelemol TMEB-35 (Trimethylolethane Tribenzoate), Pelemol PTB-35 (Pentaerythyl Tetrabenzoate), Pelemol GTO (Glyceryl Trioctanoate), Alzo International Dermol DPG-2B (Dipropylene glycol dibenzoate), Dermol B-246 (Benzyl Laurate Myristate/Palmitate), Eastman Chemical Corporation TXIB (2,2,4-trimethyl-10 1,3-pentanediol diisobutyrate), SAIB (Sucrose acetate isobutyrate), Ferro Santicizer 160 (Butyl Benzyl Phthalate), Velsicol Chemical Corporation Benzoflex 284 (Propylene glycol dibenzoate, Dipropylene glycol dibenzoate, propylene glycol monobenzoate) Benzoflex 354 (2,2,4-trimethyl-1,3-pentanediol dibenzoate), Velate 368 (2-ethylhexyl benzoate), CasChem DIPA (diisopropyl 15 adipate).

For some embodiments, plasticizers include Unitex Chemical Corporation Uniplex PX5-115 (N-Alkyl Toluenesulfonamide), Phoenix Chemical Inc. Pelemol B66 (Benzyl Benzoate), Eastman Chemical Corporation Triacetin (Glyceryl triacetate), Uniplex 82 Acetyl Tributyl Citrate.

20 The nail enamel compositions of the present invention include one or more solvents such as those generally used in conventional nail enamel compositions. Examples of these solvents include ethyl acetate, methyl acetate, n-butyl acetate, iso-butyl acetate, propyl acetate, iso-propyl acetate, amyl acetate, ethanol, isopropanol, n-amyl alcohol, n-butyl alcohol, iso-butanol, 25 cellosolve, butyl cellosolve acetate, cellosolve acetate, methyl cellosolve acetate, butyl cellosolve, ethyl cellosolve, n-butanol, xylene, aromatic (containing phenyl groups), ethers, ketones for example acetone, methyl ethyl ketone, alkanes for example, pentane, cyclopentane, hexane, heptane, cyclohexane, cyclic ethers for example, tetrahydrofuran and 1,4-dioxane, phenylated solvents for example, 30 xylene, chlorinated hydrocarbons for example, methylene chloride, chloroform, glycol ethers, N-methyl pyrrolidone alkyl lactate, and methylchloroform. It is also contemplated that toluene, if desired, can be included as a solvent or diluent

for use in a nail enamel composition in accordance with the compositions of the present invention.

The aforementioned solvents can be used alone or in mixtures thereof. In general, the amount of solvent used in the compositions of the present invention 5 range from about 53 to 82% by weight, and, for some embodiments about 65 to 75% by weight. For some embodiments, organic solvents are selected from alcohols and esters having between one and about twenty-five carbon atoms. For some embodiments, alcohols are monohydric. It is also contemplated that, if desired, monohydric alcohols are selected from ethanol, iso-propanol, and n- 10 propanol. For some embodiments, esters are selected from ethyl acetate and butyl acetate. For other embodiments, it is contemplated that polyhydric alcohols are usable.

The following are examples and Laboratory test results of enamels made according to the invention. These examples and test results are presented in 15 order to illustrate features of particular embodiments of the present invention and are not intended to limit the scope of the present invention.

EXAMPLE 1—Table 1

Clear Topcoat	1A	1B	1C
Ingredient	Weight percent	Weight percent	Weight percent
Ethyl Acetate	40.00	40.00	40.00
Butyl Acetate	21.00	21.00	21.00
Nitrocellulose (dry)	14.00	14.00	14.00
Isopropyl alcohol	6.00	6.00	6.00
Polyester resin (75% solids)	12.00	12.00	12.00
Dibutyl phthalate	7.00	--	--
Butylphthalimide			
Isopropylphthalimide	--	7.00	--
Acetyl tributylcitrate	--	--	7.00

20 The clear topcoat 1B is a topcoat formulation of the present invention, having a butylphthalimide isopropylphthalimide, BIP, concentration of 7.00 percent by weight. The clear topcoat 1A is a control, having a dibutyl phthalate component. The clear topcoat 1C is a control, having an acetyl tributylcitrate component.

Comparative mechanical film properties of Example 1 of the present invention were measured using a hardness test, adhesion test, and flexibility test as described herein.

5 **Hardness:** Using the Erichsen Pendulum Damping Tester Model 299/300 according to Persoz. (NFT 30-016). A standard 6.0 mils wet film thickness (W.F.T) Bird film applicator (MCD Industries) was used to apply films onto (4 ins X 6 ins) clean glass plates. The tests were conducted on the films after air-drying at ambient temperature for the specified times below.

Table 2

Average # oscillations

Time interval after initial application	1A	1B	1C
2 Hrs	23	26	26
24 Hrs.	50	53	58
48 Hrs.	66	73	75
72 Hrs.	75	77	81
96 Hrs.	76	74	80
168 Hrs.	94	96	98

Adhesion: Using Cross-Cut Tape Test to meet the standards of ASTM Test Method D-3359 on 6.0 mils (W.F.T) applied to (4 ins X 6 ins) clean glass

5 panels. A standard 6.0 mils (W.F.T) Bird film applicator (MCD Industries) was used to apply films onto (4 ins X 6 ins) clean glass plates. The tests were conducted on the films after air-drying at ambient temperature for the specified times below.

10 Ratings of adhesion test results according to the classification: 5=Best; 1=worst

Table 3

Time interval after initial application	1A	1B	1C
24 Hrs.	5B	5B	5B
48 Hrs.	5B	5B	5B
72 Hrs.	5B	5B	5B
96 Hrs.	5B	5B	5B
168 Hrs.	5B	5B	5B

Flexibility: According to standards of ASTM Test Method D- 522 and D 1737.

15 A standard 6.0 mils (W.F.T) Bird film applicator (MCD Industries) was used to apply films onto clean aluminum panels (6 ins X 4 ins Mill Finish 3105 H24 supplied by Q-panel Lab Products, Cleveland, Ohio). Coated panels were

uniformly bent using the BYK-Gardner Cylindrical Mandrel. The tests were conducted on the films after air-drying at ambient temperature for the specified times below.

5

Table 4

Time interval after initial application	1A	1B	1C
24 Hrs.	Good	Good	Good
48 Hrs.	Good	Good	Good
72 Hrs.	Good	Good	Good
96 Hrs.	Good	Good	Good
168 Hrs.	Good	Good	Good

10 The mechanical tests illustrated that the topcoat formulation performance of the formulation of the present invention was at least as good, when measured in terms of hardness, adhesion, and flexibility, as the performance of the topcoat free of BIP and containing dibutyl phthalate and the topcoat free of BIP and containing acetyl tributylcitrate. The topcoat formulation of the present invention did not produce any adverse effects in users.

15 The following are examples of compositions of colored nail enamel that demonstrate embodiments under the scope of the present invention. There are other possible variations that are within the scope and hence these examples are not to be construed as limitations.

Table 5

Ingredient	EXAMPLE 2	EXAMPLE 3
	Red crème shade nail enamel	Mauve pearl shade nail enamel
	Weight percent	Weight percent
Ethyl Acetate	38.410	37.950
Butyl Acetate	25.600	25.300
Nitrocellulose (dry)	12.010	11.860
Isopropyl alcohol	5.381	5.080
Tosylamide/epoxy resin	8.532	9.080
*Butylphthalimide	7.498	6.850
Isopropylphthalimide		
Stearalkonium Hectorite	0.960	1.036
Phosphoric acid	0.017	0.018
Benzophenone-1	0.175	0.188
Iron Oxide Red	0.100	--
Iron Oxide black	0.050	--
D&C Red No. 7 Calcium Lake	0.539	--
FD&C Yellow#5 Aluminum Lake	0.234	--
Titanium Dioxide	0.324	0.515
Dimethicone	0.170	0.176
Bismuth Oxychloride	--	0.400
Mica	--	1.387
D&C Red No. 34	--	0.160

Presented below is a summary of performance of specified plasticizers when used with BIP. The BIP concentration used was between 6.80 and 7.50 percent by weight. The first table shows adhesion and hardness in a red creme shade of nail enamel, made with polyester resin B75. The Pelemol GTO formulation displayed a comparatively high hardness to the BIP formulation but a lower adhesion. The BIP in each of polyester resin B75 and tosylamide/epoxy resin E75 displayed slight cracking at 168 hours. The Triacetin and BIP, Dermol DPG-2B and BIP, Pelemol DIA and BIP, and Santicizer 160 and BIP formulations displayed an adhesion that dropped off over time and a hardness that was comparatively low up to about 48 hours, but became greater over time. These formulations did not perform as well as the BIP formulation. The GTB and BIP, TXIB and BIP, and BB and BIP formulations displayed strong

adhesion and hardness over the period of the test. A similar performance was observed when a Tosylamide/ Epoxy Resin E75 was substituted for the Polyester Resin B75. These formulations displayed cracking within 24 to 48 hours.

In another test, plasticizer performance was measured in conjunction 5 with BIP in a 75/25 weight ratio and Polyester Resin B75. The performance of the plasticizer with BIP in a 75/25 ratio was comparable to performance, as measured by adhesion and hardness, of formulations of plasticizers and full strength BIP. The performance of plasticizers in a formulation with BIP in a 75/25 ratio and Tosylamide/Epoxy Resin E75 was, in some instances, weaker 10 than for full strength BIP formulations or the formulation with BIP 75/25 and Polyester Resin B75. The Pelemol GTO displayed a very low adhesion. Plasticizers that cracked within 24 to 48 hours included Uniplex 365, Pelemol 15 GTO, Uniplex 165A, and Benzoflex 284.

In one other test, plasticizers GTB, TXIB, BB and SAIB were measured 15 in a formulation that included BIP in a 50/50 ratio and Polyester Resin B75 and with BIP in a 50/50 ratio and Tosylamide Resin E75. The hardness and adhesion of enamels made with these formulations was substantially the same as formulations made with full strength BIP. Each of these formulations displayed cracking within 24 to 48 hours. Testing of the nail enamel was performed over a 20 period of seven days. It is believed that the nail enamel formulations that include BIP remain substantially free of cracks for at least about 10 days.

EVALUATION OF PLASTICIZERS IN COMBINATION WITH BIP

Table 6

Table 7

Red crème shade	ADHESION						HARDNESS				
	24h	48h	72h	144h	168h	2h	24h	48h	72h	144h	168h
BIP	5	5	4	4	4	31	82	101	102	116	127
Dermol DPG-2B	5	5	5	5	5	38	97	106	128	141	142
Pelemol DIA	5	4	4	4	3	53	128	145	184	210	205
Santicizer 160	5	4	3	3	3	37	103	107	130	147	145
GTB	5	5	4	3	3	41	95	116	121	137	142
TXIB	4	4	4	3	3	58	128	148	151	176	172
BB	5	5	5	4	4	51	120	138	144	176	160
SAIB	3	3	2	2	2	57	149	171	182	176	213
Triacetin	5	5	5	4	4	34	71	94	92	141	137
Uniplex 365	5	5	4	3	3	58	107	118	126	142	141
Pelemol GTO	5	5	5	4	4	71	161	191	200	210	209
Uniplex 165A	3	2	2	2	2	88	140	164	174	184	181
Benzoflex 284	5	4	4	3	3	37	86	108	124	149	146
Acetyl tributyl citrate	5	5	5	5	4	33	110	128	146	152	155
Dibutyl phthalate	5	5	5	5	4	40	123	138	148	153	159

Table 8
Plasticizer/ BIP in a 75/25 ratio and Polyester Resin B75

	ADHESION						HARDNESS				
	24h	48h	72h	144h	168	2h	24h	48h	72h	144h	168h
Red crème shade											
Dermol DPG-2B	5	5	5	5	5	35	87	126	128	130	137
Pelemol DIA	3	2	2	2	2	44	116	147	154	165	155
Santicizer 160	5	5	5	5	5	37	98	131	144	155	148
Triacetin	5	5	5	5	5	37	86	93	119	133	133
Uniplex 365	5	5	5	5	5	61	136	140	147	162	168
Pelemol GTO	3	3	2	2	1	61	116	123	132	156	183
Uniplex 165A	5	5	5	5	5	83	151	168	175	180	186
Benzoflex 284	5	5	5	5	5	43	98	112	113	144	143

	ADHESION						HARDNESS				
	24h	48h	72h	144h	168h	2h	24h	48h	72h	144h	168h
Red crème shade											
Dermol DPG-2B	5	4	3	3	3	41	89	123	136	141	135
Pelemol DIA	5	4	4	3	3	36	103	147	160	169	164
Santicizer 160	5	4	4	3	3	37	92	125	143	143	134
Triacetin	5	5	2	2	2	33	75	80	108	131	121
Uniplex 365	5	5	3	2	2	47	170	114	148	160	151
Pelemol GTO	0	0	0	0	0	54	100	103	110	130	128
Uniplex 165A	4	4	2	2	2	58	156	142	175	182	190
Benzoflex 284	4	4	3	2	2	35	88	95	126	141	140

Table 9
Plasticizer/BIP in a 50/50 Ratio and Polyester Resin B75

Red crème shade	ADHESION					HARDNESS					
	24h	48h	72h	144h	168h	2h	24h	48h	72h	144h	168h
GTB	5	5	5	4	4	30	85	109	120	120	158
TXIB	5	5	4	4	4	37	97	118	121	119	131
BB	5	5	4	4	3	37	96	115	119	126	143
SAIB	5	5	3	3	3	34	105	118	139	165	167

Red crème shade	ADHESION					HARDNESS					
	24h	48h	72h	144h	168h	2h	24h	48h	72h	144h	168h
GTB	5	5	5	5	5	32	88	109	138	119	139
TXIB	5	5	5	5	4	36	94	118	128	135	143
BB	5	5	5	5	5	33	89	116	129	135	145
SAIB	5	5	5	5	5	36	103	138	148	166	174

The results for use of BIP to make a Pearl shade nail enamel are shown below. The BIP showed very good values for adhesion and hardness when used with all of the resins tested. The BIP performance was better than Uniplex when used with Polyester Resin B75. The BIP performance was better than Triacetin 5 when used with Tosylamide Epoxy Resin E75. A formulation of BIP in a 75/25 ratio and Polyester Resin B75 displayed a performance better than Pelemol GTO and Dermol DPG-2B, when used as the sole plasticizer. A 50/50 ratio of BIP with Polyester Resin B75 in one embodiment, and Tosylamide Epoxy Resin E75, in another embodiment, displayed results comparable to adhesion and 10 hardness results shown with GTB, TXIB, BB and SAIB.

Table 10

Pearl shade	ADHESION					HARDNESS					
	24h	48h	72h	144h	168h	2h	24h	48h	72h	144h	168h
BIP	5	5	5	5	5	37	82	118	120	138	146
GTB	5	5	5	5	5	43	104	102	128	144	143
TXIB	4	4	3	3	3	74	130	143	154	193	183
BB	5	5	5	5	5	64	140	157	154	175	168
SAIB	4	4	4	4	4	69	162	179	168	204	210
Dermol DPG-2B	5	4	3	3	2	33	86	86	118	143	137
Pelemol DIA	5	4	3	3	2	40	112	111	153	186	176
Santicizer 160	5	4	3	3	2	45	114	125	132	156	166
Triacetin	5	5	5	5	5	35	83	108	122	147	143
Uniplex 365	5	5	5	5	5	66	128	160	149	157	168
Pelemol GTO	5	5	5	4	4	74	148	200	213	200	219
Uniplex 165A	2	2	2	2	2	88	143	154	148	176	159
Benzoflex 284	5	5	5	5	5	34	83	101	133	154	155
Acetyl tributyl citrate	5	5	5	5	5	42	108	129	154	159	160
Dibutyl phthalate	5	5	5	5	5	48	120	131	159	159	158

Pearl shade	Plasticizers and Polyester Resin B75				
	24h	48h	72h	144h	168h
BIP	5	5	5	5	5
GTB	5	5	5	5	5

Pearl shade	Plasticizers and Tosylamide/ Epoxy Resin E75				
	24h	48h	72h	144h	168h
BIP	27	78	101	112	129
GTB	32	98	116	118	113

TXIB	5	4	4	4	4	52	130	139	145	170	178
BB	5	5	5	5	5	48	117	138	141	160	168
SAIB	5	4	4	4	4	56	154	177	173	217	202
Dermol DPG-2B	5	5	5	4	4	32	74	86	119	134	138
Pelemol DIA	5	4	3	3	3	42	120	169	176	176	162
Santicizer 160	5	4	4	4	3	44	117	130	153	163	171
Triacetin	2	2	2	2	2	29	73	92	113	149	143
Uniplex 365	2	2	2	2	2	53	123	132	147	169	168
Pelemol GTO	5	5	5	4	4	74	148	200	213	200	209
Uniplex 165A	5	5	4	3	3	89	183	189	189	192	202
Benzoflex 284	5	5	5	5	5	34	83	101	133	154	155
Acetyl tributyl citrate	5	5	5	5	5	33	110	128	146	152	155
Dibutyl phthalate	5	5	5	5	4	40	123	138	148	153	159

Table 11
Plasticizer/BIP in a 75/25 Ratio with Polyester Resin B75

Pearl shade	Plasticizer/BIP in a 75/25 Ratio with Polyester Resin B75			
Dermol DPG-2B	5	4	3	2
Pelemol DIA	5	4	2	3
Santicizer 160	5	4	4	3
Triacetin	5	5	5	5
Uniplex 365	5	5	5	5
Pelemol GTO	3	3	2	2
Uniplex 165A	5	5	5	5
Benzoflex 284	5	5	5	5

Plasticizer/BIP in a 75/25 Ratio with Tosylamide/Epoxy Resin E75

Pearl shade	Plasticizer/BIP in a 75/25 Ratio with Tosylamide/Epoxy Resin E75			
Dermol DPG-2B	5	5	4	4
Pelemol DIA	5	4	4	3
Santicizer 160	5	5	5	5
Triacetin	5	5	5	5
Uniplex 365	5	5	5	5
Pelemol GTO	5	5	5	5
Uniplex 165A	5	5	5	4.5
Benzoflex 284	5	5	5	5

Pearl shade	Plasticizer/BIP in a 50/50 Ratio with Polyester Resin B75			
Dermol DPG-2B	5	5	3	3
Pelemol DIA	5	5	4	34
Santicizer 160	5	5	5	35
Triacetin	5	5	5	31
Uniplex 365	5	5	5	49
Pelemol GTO	5	5	5	52
Uniplex 165A	5	5	5	65
Benzoflex 284	5	5	5	33

Plasticizer/BIP in a 50/50 Ratio with Tosylamide Epoxy Resin E75						
Pearl shade	5	5	5	4	3	41
GTB	5	5	5	4	4	37
TXIB	5	5	4	4	4	97
BB	5	5	4	4	3	37
SAIB	5	5	3	3	3	96
						100
						122
						121
						133
						158
						118
						121
						119
						131
						115
						119
						126
						143
						165
						167
						118
						139
						165
						167

Additional examples using Pelemol BIP in combination with Glyceryl Triacetate

Table 12

Red Crème shade	FORMULA EC	FORMULA FC	FORMULA GC	FORMULA HC ¹	FORMULA IC ²
Ethyl Acetate	41.271	41.238	41.386	41.262	41.262
Butyl Acetate	22.234	22.200	22.282	22.218	22.218
Nitrocellulose (dry) ¼ sec R.S	9.603	9.603	6.005	9.603	9.603
½ sec R.S	2.407	2.407	6.005	2.407	2.407
Isopropyl alcohol	5.380	5.380	5.150	5.15	5.15
Tosylamide/epoxy resin	8.532	7.644	7.644	7.644	8.880
Butylphthalimide isopropylphthalimide	7.498	7.498	7.498	2.983	4.488
Acrylic- silicone copolymer	0.000	0.955	0.955	0.955	0.955
Glyceryl Triacetate	0.000	0.000	0.000	4.516	3.010
Stearalkonium Hectorite	0.960	0.960	0.960	0.960	0.960
Benzophenone-1	0.175	0.175	0.175	0.175	0.175
Iron Oxides	0.153	0.153	0.153	0.153	0.153
Phosphoric acid	0.017	0.017	0.017	0.017	0.017
D&C Red No.6	0.673	0.673	0.673	0.673	0.673
D&C Red No. 7 Calcium Lake	0.539	0.539	0.539	0.539	0.539
Titanium Dioxide	0.324	0.324	0.324	0.324	0.324
FD&C Yellow No. 5 Aluminum Lake	0.234	0.234	0.234	0.234	0.234

ratios of BIP/ Glyceryl Triacetate

C = crème shade

(1) Formula HC: Ratio of BIP/GTA - 40/60

(2) Formula IC: Ratio BIP/GTA- 60/40

(3) Formula HP: Ratio of BIP/GTA 30/70

(4) Formula IP: Ratio of BIP/GTA 50/50

P = Pearl shade

Table 13 TEST RESULTS Red Crème shade

ratios of BIP/ Glyceryl Triacetate

Table 14

Pearl shade	FORMULA EP	FORMULA FP	FORMULA GP	FORMULA HP ³	FORMULA HP ⁴
Ethyl Acetate	40.581	40.576	40.576	40.576	40.576
Butyl Acetate	21.853	21.849	21.849	21.849	21.849
Nitrocellulose (dry) 1/4 sec R.S 1/2 sec R.S	11.858	11.858	5.929	11.858	11.858
Isopropyl alcohol	5.082	5.082	5.082	5.082	5.082
Tosylamide/epoxy resin (solids)	9.081	8.081	8.081	8.081	8.081
Butylphthalimide isopropylphthalimide	6.854	6.854	6.854	2.081	3.427
Acrylic- silicone copolymer solids)	0.000	1.009	1.009	1.009	1.009
Glyceryl Triacetate	0.000	0.000	0.000	4.772	3.427
Stearalkonium Hectorite	1.036	1.036	1.036	1.036	1.036
Bismuth Oxychloride	0.40	0.40	0.40	0.40	0.40
Dimethicone	0.176	0.176	0.176	0.176	0.176
Iron Oxides	0.811	0.811	0.811	0.811	0.811
Benzophenone-1	0.188	0.188	0.188	0.188	0.188
Phosphoric acid	0.018	0.018	0.018	0.018	0.018
Mica	1.387	1.387	1.387	1.387	1.387
D&C Red No. 34	0.160	0.160	0.160	0.160	0.160
Titanium Dioxide	0.515	0.515	0.515	0.515	0.515

Table 15
TEST RESULTS

	Pearl shade					
	FORMULA EP	FORMULA FP	FORMULA GP	FORMULA HP ³	FORMULA IP ⁴	FORMULA IP ⁴
<u>Hardness:</u>						
2Hrs.	21	25	24	21	22	
24 Hrs.	64	64	63	49	51	
48 Hrs.	76	84	81	64	68	
72 Hrs.	99	98	93	74	77	
144 Hrs.	126	121	126	109	108	
168 Hrs.	128	140	139	125	126	
<u>Adhesion:</u>						
24 Hrs.	5	5	5	5	5	5
48Hrs.	5	5	5	5	5	5
72 Hrs.	5	5	5	5	5	5
144 Hrs.	5	5	5	5	5	5
168 Hrs.	5	5	5	5	5	5
<u>Flexibility:</u>						
24 Hrs.	Ok	Ok	Ok	Ok	Ok	Ok
48Hrs.	Ok	Ok	Ok	Ok	Ok	Ok
72 Hrs.	Ok	Ok	Ok	Ok	Ok	Ok
144 Hrs.	Stretched	Stretched	Stretched	Stretched	Ok	Ok
168 Hrs.	Sl. Cracked	Sl. Cracked	Sl. Cracked	Sl. Cracked	Ok	Ok

Testing of the red creme shade and the pearl shade was performed to confirm that BIP formulations are usable in formulations containing a variety of types and concentrations of coloring agents. The red creme formulations displayed lesser adhesion and flexibility than pearl shade formulations. This 5 performance parallels a tendency for the red creme shade to chip more easily in phthalate-based formulations.

The nail enamel embodiments of the present invention are made by blending the BIP into the formulation alone, or, optionally, with other plasticizers. Other ingredients are added in accordance with methods known to 10 those skilled in the art. The nail enamel is dispensed into containers. The containers typically have applicators that are an integral part of the container or that are packaged with the container. One type of applicator is a brush. The enamel is applied to nails as a base coat or a topcoat.

A consumer use study was conducted on the mauve pearl shade. The 15 study included seven test subjects. The two products tested included a formulation that included BIP and that was phthalate-free and a formulation that included phthalates. There was no difference detected between the two products for overall wear and chipping. Gloss and appearance were also comparable, although a preference for the gloss of the BIP containing formula was noted in 20 the laboratory.

Each of any foregoing patents, patent applications and references described herein is incorporated by reference in its entirety. Having described particular embodiments in accordance with the present invention, it is believed that other modifications, variations and changes will be suggested to those 25 skilled in the art in view of the teachings set forth herein. It is, therefore, to be understood that all such variations, modifications, and changes are believed to fall within the scope of the present invention as defined by the appended claims.

IN THE CLAIMS

What is claimed is:

- 5 1. A cosmetic film, comprising butylphthalimide isopropylphthalimide; one or more primary film forming polymers and one or more secondary film forming resins.
- 10 2. The cosmetic film of claim 1, further comprising a modifying plasticizer.
- 15 3. The cosmetic film of claim 1 wherein the secondary film-forming polymers are selected from the group consisting of polyurethanes, polyacryls, polymethacryls, cellulosic polymers, styrene-acryl copolymers, polystyrene-polyacryl mixtures, polysiloxanes, polyesters, polyethers, cellulose acetate propionate, urethane-acryl copolymers, siloxane-urethane copolymers, vinyl acetate polymers, and mixtures thereof.
- 20 4. The cosmetic film of claim 1 wherein the secondary film forming polymers are selected from a group consisting of acrylic-silicone resin, polyester resin, and polyamide resin.
- 25 5. The cosmetic film of claim 2 wherein the modifying plasticizer is selected from the group consisting of N-ethyl-o/p-Toluenesulfonamide, di-(2-ethylhexyl)adipate, diisobutyladipate, glyceryl tribenzoate, pentaerytritol tetrabenzoate, diisopropyl adipate, trimethylolethane tribenzoate, pentaerythyl tetrabenzoate, glyceryl trioctanoate, dipropylene glycol dibenzoate, benzyl laurate myristate/palmitate, 2,2,4-trimethyl-1,3-pentanediol diisobutyrate, sucrose acetate isobutyrate, butyl benzyl phthalate, propylene glycol dibenzoate, dipropylene glycol dibenzoate, propylene glycol monobenzoate, 2,2,4-trimethyl-1,3-pentanediol dibenzoate, 2-

ethylhexyl benzoate, diisopropyl adipate, N-alkyl toluene sulfonamide, benzyl benzoate, glyceryl triacetate, and acetyl tributyl citrate.

5 6. The cosmetic film of claim 1, further comprising one or more solvents.

10 7. The cosmetic film of claim 6 wherein the solvent is selected from a group consisting of ethyl acetate, methyl acetate, n-butyl acetate, iso-butyl acetate, propyl acetate, iso-propyl acetate, amyl acetate, ethanol, isopropanol, n-amyl alcohol, n-butyl alcohol, iso-butanol, cellosolve, butyl cellosolve, ethyl cellosolve, n-butanol, xylene, aromatic ethers, ketones, methyl ethyl ketone, alkanes, cyclopentane, hexane, heptane, cyclohexane, cyclic ethers, phenylated solvents, chlorinated hydrocarbons, and toluene.

15 8. A nail enamel, comprising: butylphthalimide isopropylphthalimide; a secondary film forming polymer; nitrocellulose; and a polyester resin.

20 9. A colored nail enamel, consisting essentially of butylphthalimide isopropylphthalimide in a concentration of about 1 to 10 percent by weight; ethyl acetate in a concentration of about 37 to 39 percent by weight; butyl acetate in a concentration of about 19 to 26 percent by weight; nitrocellulose in a concentration of about 11 to 14 percent by weight; isopropyl alcohol in a concentration of about 5 to 6 percent by weight; optionally, polyester resin in a concentration of about 12 percent by weight; optionally, tosylamide/epoxy resin in a concentration of about 8 to 10 percent by weight; stearalkonium hectorite in a concentration of about 0.5 to 2 percent by weight; optionally, acrylic copolymer in a concentration of about 1.5 percent by weight; and coloring agents in a concentration effective to produce color.

25

30

10. A nail enamel, comprising: a plasticizer and butylphthalimide isopropylphthalimide (BIP) in a concentration effective for imparting to the nail enamel, adhesion, hardness and flexibility that is retained for at least about 7 days, when the nail enamel is applied to a nail.
5
11. The nail enamel of claim 10, wherein the resin is either a tosylamide/epoxy resin or a polyester resin.
- 10 12. The nail enamel of claim 10 wherein the nail enamel is substantially free of cracking for at least about 7 days after application to a nail.
- 15 13. The nail enamel of claim 1 wherein the enamel is substantially free of phthalates.
14. A colored nail enamel, comprising: ethyl acetate; butyl acetate; nitrocellulose; isopropyl alcohol; tosylamide/epoxy resin; butylphthalimide isopropylphthalimide in a concentration of about 1 to about 10 percent by weight; and coloring agents.
20
15. A method for making a cosmetic film and that has a hardness, adhesion, flexibility, gloss and long shelf life that prevent cracking for at least about 7 days after application to a nail, comprising: providing one or more primary film forming polymers; providing one or more secondary film forming resins; providing butylphthalimide isopropylphthalimide; and blending the primary film forming polymer, secondary film forming resin and butylphthalimide isopropylphthalimide in a weight percent concentration of about 1 to 10 weight percent to make a cosmetic film having a hardness, adhesion, flexibility, gloss and long shelf life.
25
- 30

16. The method of claim 15, further comprising blending the butylphthalimide isopropylphthalimide with another plasticizer.
17. The method of claim 15, further comprising adding color agents to the cosmetic film.
18. A method for coating nails with a cosmetic film, comprising: providing a cosmetic film comprising butylphthalimide isopropylphthalimide; and applying the cosmetic film to one or more mammalian nail.
19. A colored nail enamel, comprising:
An acetate;
Nitrocellulose;
butylphthalimide isopropylphthalimide;
tosylamide/epoxy resin; and
coloring agents.
20. The colored nail enamel of claim 19 wherein the butylphthalimide isopropylphthalimide concentration is within a range of about 1 and 10 percent by weight.
21. A cosmetic film, comprising butylphthalimide isopropylphthalimide.
22. A cosmetic film, comprising butylphthalimide.
23. A cosmetic film, comprising isopropylphthalimide.

INTERNATIONAL SEARCH REPORT

International application No.
PCT/US02/16177

A. CLASSIFICATION OF SUBJECT MATTER

IPC(7) :A61K 7/00, 7/04

US CL :424/61, 400, 401

According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)

U.S. : 424/61, 400, 401

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched
NONE

Electronic data base consulted during the international search (name of data base and, where practicable, search terms used)

STN, CAS ONLINE, WEST

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
Y	US 5,792,447 B1(SOCCI ET AL.) 11 AUGUST 1998, See entire document.	1-23
Y	US 5,275,807, B1(PAPPAS ET AL.) 04 JANUARY 1994, See entire document.	1-23
Y	US 4,421,881 B1(BENKENDORF ET AL.) 20 DECEMBER 1983, See entire document.	1-23

 Further documents are listed in the continuation of Box C. See patent family annex.

Special categories of cited documents:			
"A"	document defining the general state of the art which is not considered to be of particular relevance	"T"	later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention
"E"	earlier document published on or after the international filing date	"X"	document of particular relevance; the claimed invention cannot be considered novel or cannot be considered to involve an inventive step when the document is taken alone
"L"	document which may throw doubts on priority claim(s) or which is cited to establish the publication date of another citation or other special reason (as specified)	"V"	document of particular relevance; the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art
"O"	document referring to an oral disclosure, use, exhibition or other means	"G"	document member of the same patent family
"P"	document published prior to the international filing date but later than the priority date claimed		

Date of the actual completion of the international search	Date of mailing of the international search report
26 JULY 2002	19 SEP 2002
Name and mailing address of the ISA/US Commissioner of Patents and Trademarks Box PCT Washington, D.C. 20231 Facsimile No. (703) 305-8230	Authorized officer <i>Valerie Bell-Thomason</i> SHELLEY A. DODSON Telephone No. (703) 308-1235

**This Page is Inserted by IFW Indexing and Scanning
Operations and is not part of the Official Record**

BEST AVAILABLE IMAGES

Defective images within this document are accurate representations of the original documents submitted by the applicant.

Defects in the images include but are not limited to the items checked:

- BLACK BORDERS**
- IMAGE CUT OFF AT TOP, BOTTOM OR SIDES**
- FADED TEXT OR DRAWING**
- BLURRED OR ILLEGIBLE TEXT OR DRAWING**
- SKEWED/SLANTED IMAGES**
- COLOR OR BLACK AND WHITE PHOTOGRAPHS**
- GRAY SCALE DOCUMENTS**
- LINES OR MARKS ON ORIGINAL DOCUMENT**
- REFERENCE(S) OR EXHIBIT(S) SUBMITTED ARE POOR QUALITY**
- OTHER: _____**

IMAGES ARE BEST AVAILABLE COPY.

As rescanning these documents will not correct the image problems checked, please do not report these problems to the IFW Image Problem Mailbox.